Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# (*E*)-*N*′-(3,4-Dihydroxybenzylidene)-2,4-dimethylbenzohydrazide monohydrate

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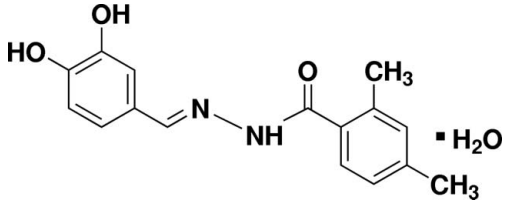
Received 18 February 2013; accepted 27 February 2013

Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.040; $wR$ factor = 0.111; data-to-parameter ratio = 13.1.

In the title compound, $C_{16}H_{16}N_2O_3\cdot H_2O$, the dihedral angle between the benzene rings is 30.27 (7)°. In the crystal, the components are linked by N—H···O, O—H···O and C—H···O interactions into a three-dimensional network.

## Related literature

For the applications and biological activity of Schiff bases, see: Musharraf *et al.* (2012); Khan *et al.* (2012). For the crystal structures of related compounds, see: Taha *et al.* (2012); Baharudin *et al.* (2012).




## Experimental

### Crystal data

$C_{16}H_{16}N_2O_3\cdot H_2O$
$M_r$ = 302.32
Monoclinic, $P2_1/n$
$a$ = 8.1373 (3) Å
$b$ = 13.9025 (5) Å
$c$ = 13.7886 (5) Å
$\beta$ = 92.913 (1)°
$V$ = 1557.87 (10) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 298 K
0.30 × 0.10 × 0.10 mm

### Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min}$ = 0.973, $T_{max}$ = 0.991
9012 measured reflections
2897 independent reflections
2535 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.016

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.111
$S$ = 1.05
2897 reflections
221 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1$W$—H1$W$1···N2$^{i}$ | 0.87 (2) | 2.20 (2) | 3.059 (2) | 169 (2) |
| O1$W$—H2$W$1···O1 | 0.94 (2) | 2.01 (2) | 2.935 (2) | 173 (2) |
| N1—H1$A$···O3$^{ii}$ | 0.91 (2) | 2.08 (2) | 2.962 (2) | 163 (2) |
| O2—H2$A$···O1$^{i}$ | 0.88 (2) | 1.94 (2) | 2.791 (2) | 162 (2) |
| O3—H3$A$···O1$W^{iii}$ | 0.85 (2) | 1.79 (2) | 2.629 (2) | 172 (2) |
| C8—H8$A$···O3$^{ii}$ | 0.93 | 2.58 | 3.382 (2) | 145 |
| C15—H15$B$···O2$^{i}$ | 0.96 | 2.52 | 3.351 (2) | 144 |

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).

The authors thank the Research Management Institute of UiTM for financial support under the Dana Kecemerlangan Grant Scheme [grant No. 600-RMI/DANA 5/3 RIF (143/2012)].

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2622).

## References

Baharudin, M. S., Taha, M., Ismail, N. H., Shah, S. A. A. & Yousuf, S. (2012). *Acta Cryst.* E**68**, o3255.
Bruker (2000). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Khan, K. M., Taha, M., Naz, F., Siddiqui, S., Rahim, F., Perveen, S. & Choudhary, M. I. (2012). *Med. Chem.* **8**, 705–710.
Musharraf, S. G., Bibi, A., Shahid, N., Najam-ul-Haq, M., Khan, M., Taha, M., Mughal, U. R. & Khan, K. M. (2012). *Am. J. Anal. Chem.* **3**, 779-789.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Taha, M., Naz, H., Rahman, A. A., Ismail, N. H. & Sammer, Y. (2012). *Acta Cryst.* E**68**, o2778.

# supporting information

*Acta Cryst.* (2013). E**69**, o490 [doi:10.1107/S1600536813005692]

## (*E*)-*N*′-(3,4-Dihydroxybenzylidene)-2,4-dimethylbenzohydrazide monohydrate

**Muhammad Taha, Nor Hadiani Ismail, Faridahanim Mohd Jaafar, Ahmad Nazif Aziz and Sammer Yousuf**

### S1. Comment

Structurally diverse range of benzohydrazides have been extensively studied in order to explore the structural features that may be responsible for different biological activities (Musharraf *et al.*, 2012; Khan *et al.*, 2012). The title compound is yet another benzohydrazide monohydrate, obtained as a part of our ongoing research that has been studied by X-ray crystallographic method and reported in this article.

In the title compound (Fig. 1) dimethyl and dihydroxy substituted benzene rings (C1–C6 and C9–C14, respectively) are each planner with a dihedral angle 30.27 (7)° between their mean-planes. The azomethine double bond, N2═C8 (1.2729 (19) Å) adopts an *E* configuration. The bond lengths and angle are similar to the corresponding bond lengths and angles reported in structurally related benzohydrazide derivatives (Taha *et al.*, 2012; Baharudin *et al.*, 2012). The crystal structure is stabilized by N1—H1A···N2, O2—H2A···O1, C8—H8A···O3 and C15—H15B···O2 intermolecular interactions. The ineteractions further extend the structure to a three dimentional network *via* O1W—H2W1···O1, O1W —H1A···O3 and O3—H3A···O1W interactions involving the water of hydration (Table 2 and Fig. 2).

### S2. Experimental

The title compound was synthesized by reacting (0.328 g, 2 mmol) 2,4-dimethylbenzohydrazide and (0.276 g, 2 mmol) 3,4-dihydroxybenzaldehyde as starting meterial under the same conditions and solvents as described previously for the synthesis of benzohydrazides (Taha *et al.*, 2012). The title compound was recrystalized by dissolving in methanol to obtain colorless needles (0.499 g, 88% yield). All chemicals were purchased by Sigma Aldrich Germany.

### S3. Refinement

H atoms on methyl and benzene ring were positioned geometrically with C—H = 0.96 and 0.93 Å, respectively and constrained to ride on their parent atoms with $U_{iso}$(H) = 1.2$U_{eq}$(benzene) or 1.5$U_{eq}$(methyl). The H atoms on oxygen and nitrogen were located in difference Fourier map and refined isotropically. A rotating group model was applied to the methyl groups.

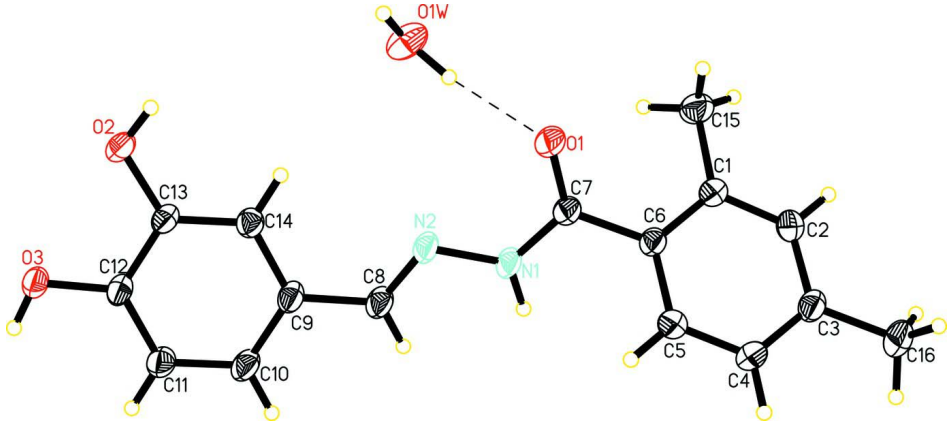



**Figure 1**

The molecular structure of the title compound with displacement ellipsoids drawn at 30% probability level.

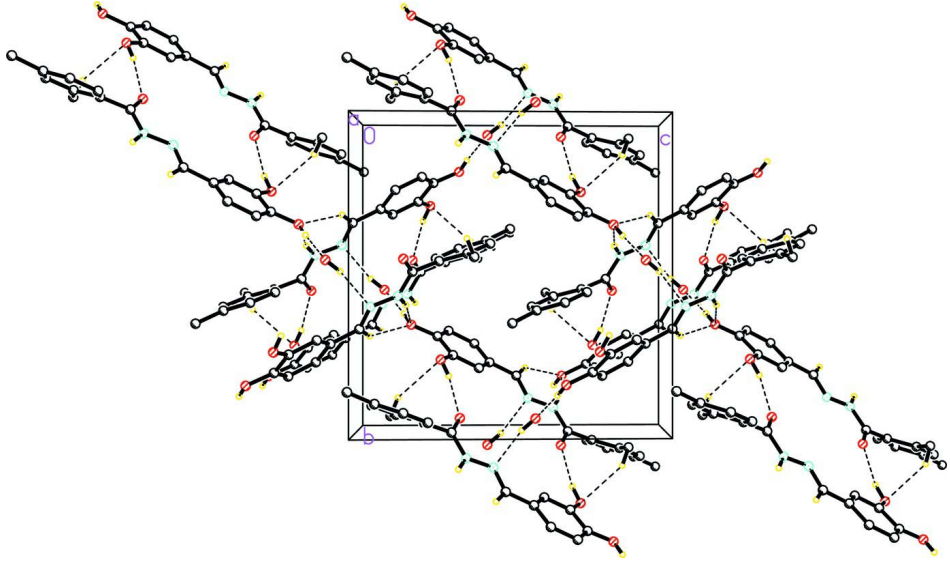



**Figure 2**

The crystal packing of the title compound. Only hydrogen atoms involved in hydrogen bonding are shown.

### (*E*)-*N*′-(3,4-Dihydroxybenzylidene)-2,4-dimethylbenzohydrazide monohydrate

*Crystal data*

$C_{16}H_{16}N_2O_3·H_2O$
$M_r$ = 302.32
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a$ = 8.1373 (3) Å
$b$ = 13.9025 (5) Å
$c$ = 13.7886 (5) Å
$\beta$ = 92.913 (1)°
$V$ = 1557.87 (10) Å$^3$
$Z$ = 4
$F(000)$ = 640
$D_x$ = 1.289 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 4856 reflections
$\theta$ = 2.8–28.2°
$\mu$ = 0.09 mm$^{-1}$
$T$ = 298 K
Block, brown
0.30 × 0.10 × 0.10 mm

*Data collection*

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min}$ = 0.973, $T_{max}$ = 0.991
9012 measured reflections
2897 independent reflections
2535 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.016
$\theta_{max}$ = 25.5°, $\theta_{min}$ = 2.1°
$h$ = −9→9
$k$ = −16→16
$l$ = −16→13

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.111
$S$ = 1.05
2897 reflections
221 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.3352P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted $R$-factor $wR$ and goodness of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating $R$-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.15363 (13) | 0.55077 (8) | −0.16328 (8) | 0.0569 (3) |
| O1W | −0.12355 (15) | 0.45259 (10) | −0.07769 (9) | 0.0612 (3) |
| O2 | −0.04800 (12) | 0.26761 (9) | 0.22605 (8) | 0.0505 (3) |
| O3 | 0.16469 (13) | 0.15980 (8) | 0.32684 (7) | 0.0460 (3) |
| N1 | 0.34634 (15) | 0.43523 (9) | −0.14057 (9) | 0.0454 (3) |
| N2 | 0.28104 (14) | 0.40196 (9) | −0.05580 (8) | 0.0435 (3) |
| C1 | 0.30375 (16) | 0.56844 (10) | −0.36100 (10) | 0.0395 (3) |
| C2 | 0.40413 (18) | 0.60048 (11) | −0.43273 (10) | 0.0446 (3) |
| H2C | 0.3561 | 0.6153 | −0.4935 | 0.054* |
| C3 | 0.57324 (17) | 0.61142 (11) | −0.41767 (10) | 0.0435 (3) |
| C4 | 0.64410 (17) | 0.58608 (11) | −0.32796 (11) | 0.0455 (4) |
| H4A | 0.7574 | 0.5911 | −0.3166 | 0.055* |
| C5 | 0.54846 (16) | 0.55358 (10) | −0.25556 (10) | 0.0423 (3) |
| H5A | 0.5981 | 0.5371 | −0.1956 | 0.051* |
| C6 | 0.37828 (16) | 0.54484 (9) | −0.27016 (10) | 0.0366 (3) |

| | | | | |
|---|---|---|---|---|
| C7 | 0.28131 (16) | 0.51150 (10) | −0.18789 (10) | 0.0397 (3) |
| C8 | 0.35766 (18) | 0.33077 (11) | −0.01702 (11) | 0.0464 (4) |
| H8A | 0.4495 | 0.3069 | −0.0465 | 0.056* |
| C9 | 0.30656 (17) | 0.28538 (10) | 0.07190 (10) | 0.0421 (3) |
| C10 | 0.41516 (19) | 0.22570 (11) | 0.12370 (12) | 0.0514 (4) |
| H10A | 0.5192 | 0.2145 | 0.1011 | 0.062* |
| C11 | 0.37047 (19) | 0.18256 (11) | 0.20869 (12) | 0.0499 (4) |
| H11A | 0.4448 | 0.1431 | 0.2432 | 0.060* |
| C12 | 0.21552 (16) | 0.19792 (9) | 0.24262 (10) | 0.0390 (3) |
| C13 | 0.10379 (16) | 0.25680 (10) | 0.18973 (10) | 0.0376 (3) |
| C14 | 0.14912 (16) | 0.29972 (10) | 0.10553 (10) | 0.0392 (3) |
| H14A | 0.0745 | 0.3387 | 0.0705 | 0.047* |
| C15 | 0.12199 (18) | 0.55713 (14) | −0.38483 (12) | 0.0571 (4) |
| H15A | 0.1029 | 0.5506 | −0.4538 | 0.086* |
| H15B | 0.0648 | 0.6128 | −0.3628 | 0.086* |
| H15C | 0.0825 | 0.5008 | −0.3531 | 0.086* |
| C16 | 0.6765 (2) | 0.65068 (14) | −0.49624 (13) | 0.0615 (5) |
| H16A | 0.6324 | 0.6293 | −0.5584 | 0.092* |
| H16B | 0.7875 | 0.6280 | −0.4863 | 0.092* |
| H16C | 0.6755 | 0.7197 | −0.4941 | 0.092* |
| H2A | −0.099 (3) | 0.3180 (16) | 0.1996 (15) | 0.077 (6)* |
| H1A | 0.436 (2) | 0.4053 (13) | −0.1634 (13) | 0.061 (5)* |
| H1W1 | −0.165 (3) | 0.4883 (17) | −0.0335 (16) | 0.080 (7)* |
| H3A | 0.239 (2) | 0.1248 (15) | 0.3533 (15) | 0.071 (6)* |
| H2W1 | −0.029 (3) | 0.4798 (17) | −0.1022 (16) | 0.091 (7)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0495 (6) | 0.0633 (7) | 0.0601 (7) | 0.0209 (5) | 0.0261 (5) | 0.0182 (5) |
| O1W | 0.0486 (6) | 0.0766 (9) | 0.0595 (7) | −0.0124 (6) | 0.0122 (5) | −0.0274 (6) |
| O2 | 0.0399 (5) | 0.0578 (7) | 0.0557 (7) | 0.0085 (5) | 0.0200 (5) | 0.0140 (5) |
| O3 | 0.0453 (6) | 0.0499 (6) | 0.0442 (6) | 0.0056 (5) | 0.0153 (5) | 0.0141 (5) |
| N1 | 0.0462 (6) | 0.0506 (7) | 0.0414 (7) | 0.0134 (6) | 0.0227 (5) | 0.0112 (5) |
| N2 | 0.0453 (6) | 0.0477 (7) | 0.0393 (6) | 0.0066 (5) | 0.0194 (5) | 0.0077 (5) |
| C1 | 0.0360 (7) | 0.0429 (7) | 0.0400 (7) | 0.0004 (5) | 0.0051 (6) | 0.0011 (6) |
| C2 | 0.0478 (8) | 0.0505 (8) | 0.0358 (7) | 0.0008 (6) | 0.0041 (6) | 0.0062 (6) |
| C3 | 0.0437 (7) | 0.0430 (8) | 0.0448 (8) | −0.0010 (6) | 0.0136 (6) | 0.0037 (6) |
| C4 | 0.0335 (7) | 0.0506 (8) | 0.0530 (9) | −0.0033 (6) | 0.0068 (6) | 0.0038 (7) |
| C5 | 0.0383 (7) | 0.0488 (8) | 0.0396 (7) | 0.0023 (6) | 0.0014 (6) | 0.0048 (6) |
| C6 | 0.0360 (7) | 0.0379 (7) | 0.0367 (7) | 0.0023 (5) | 0.0085 (5) | 0.0023 (5) |
| C7 | 0.0378 (7) | 0.0434 (7) | 0.0387 (7) | 0.0050 (6) | 0.0105 (6) | 0.0030 (6) |
| C8 | 0.0484 (8) | 0.0466 (8) | 0.0463 (8) | 0.0108 (7) | 0.0218 (6) | 0.0064 (7) |
| C9 | 0.0468 (8) | 0.0393 (7) | 0.0418 (7) | 0.0055 (6) | 0.0176 (6) | 0.0046 (6) |
| C10 | 0.0468 (8) | 0.0526 (9) | 0.0572 (9) | 0.0149 (7) | 0.0263 (7) | 0.0122 (7) |
| C11 | 0.0469 (8) | 0.0496 (9) | 0.0547 (9) | 0.0156 (7) | 0.0171 (7) | 0.0154 (7) |
| C12 | 0.0441 (7) | 0.0352 (7) | 0.0388 (7) | 0.0008 (6) | 0.0145 (6) | 0.0039 (5) |
| C13 | 0.0370 (7) | 0.0365 (7) | 0.0405 (7) | 0.0007 (5) | 0.0128 (5) | −0.0002 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C14 | 0.0420 (7) | 0.0361 (7) | 0.0401 (7) | 0.0036 (6) | 0.0082 (6) | 0.0034 (6) |
| C15 | 0.0401 (8) | 0.0788 (12) | 0.0521 (9) | −0.0036 (7) | −0.0010 (7) | 0.0037 (8) |
| C16 | 0.0567 (9) | 0.0699 (11) | 0.0598 (10) | −0.0030 (8) | 0.0225 (8) | 0.0146 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C7 | 1.2364 (16) | C5—C6 | 1.3945 (19) |
| O1W—H1W1 | 0.87 (2) | C5—H5A | 0.9300 |
| O1W—H2W1 | 0.94 (2) | C6—C7 | 1.4884 (18) |
| O2—C13 | 1.3645 (15) | C8—C9 | 1.4581 (19) |
| O2—H2A | 0.88 (2) | C8—H8A | 0.9300 |
| O3—C12 | 1.3599 (16) | C9—C10 | 1.384 (2) |
| O3—H3A | 0.85 (2) | C9—C14 | 1.3988 (19) |
| N1—C7 | 1.3398 (18) | C10—C11 | 1.382 (2) |
| N1—N2 | 1.3877 (15) | C10—H10A | 0.9300 |
| N1—H1A | 0.913 (19) | C11—C12 | 1.3835 (19) |
| N2—C8 | 1.2729 (19) | C11—H11A | 0.9300 |
| C1—C2 | 1.3878 (19) | C12—C13 | 1.4004 (19) |
| C1—C6 | 1.4026 (19) | C13—C14 | 1.3725 (19) |
| C1—C15 | 1.5069 (19) | C14—H14A | 0.9300 |
| C2—C3 | 1.390 (2) | C15—H15A | 0.9600 |
| C2—H2C | 0.9300 | C15—H15B | 0.9600 |
| C3—C4 | 1.384 (2) | C15—H15C | 0.9600 |
| C3—C16 | 1.507 (2) | C16—H16A | 0.9600 |
| C4—C5 | 1.373 (2) | C16—H16B | 0.9600 |
| C4—H4A | 0.9300 | C16—H16C | 0.9600 |
| | | | |
| H1W1—O1W—H2W1 | 112 (2) | C10—C9—C14 | 119.07 (13) |
| C13—O2—H2A | 110.5 (13) | C10—C9—C8 | 119.44 (12) |
| C12—O3—H3A | 110.4 (13) | C14—C9—C8 | 121.48 (13) |
| C7—N1—N2 | 121.02 (11) | C11—C10—C9 | 120.62 (13) |
| C7—N1—H1A | 119.8 (11) | C11—C10—H10A | 119.7 |
| N2—N1—H1A | 119.2 (11) | C9—C10—H10A | 119.7 |
| C8—N2—N1 | 114.37 (11) | C10—C11—C12 | 120.24 (14) |
| C2—C1—C6 | 117.91 (12) | C10—C11—H11A | 119.9 |
| C2—C1—C15 | 119.00 (13) | C12—C11—H11A | 119.9 |
| C6—C1—C15 | 123.05 (12) | O3—C12—C11 | 123.41 (13) |
| C1—C2—C3 | 122.88 (13) | O3—C12—C13 | 117.06 (11) |
| C1—C2—H2C | 118.6 | C11—C12—C13 | 119.52 (12) |
| C3—C2—H2C | 118.6 | O2—C13—C14 | 123.35 (12) |
| C4—C3—C2 | 118.05 (13) | O2—C13—C12 | 116.69 (12) |
| C4—C3—C16 | 120.85 (13) | C14—C13—C12 | 119.96 (12) |
| C2—C3—C16 | 121.10 (13) | C13—C14—C9 | 120.57 (13) |
| C5—C4—C3 | 120.53 (13) | C13—C14—H14A | 119.7 |
| C5—C4—H4A | 119.7 | C9—C14—H14A | 119.7 |
| C3—C4—H4A | 119.7 | C1—C15—H15A | 109.5 |
| C4—C5—C6 | 121.27 (13) | C1—C15—H15B | 109.5 |
| C4—C5—H5A | 119.4 | H15A—C15—H15B | 109.5 |

| | | | |
|---|---|---|---|
| C6—C5—H5A | 119.4 | C1—C15—H15C | 109.5 |
| C5—C6—C1 | 119.32 (12) | H15A—C15—H15C | 109.5 |
| C5—C6—C7 | 118.57 (12) | H15B—C15—H15C | 109.5 |
| C1—C6—C7 | 122.11 (12) | C3—C16—H16A | 109.5 |
| O1—C7—N1 | 122.17 (12) | C3—C16—H16B | 109.5 |
| O1—C7—C6 | 123.86 (12) | H16A—C16—H16B | 109.5 |
| N1—C7—C6 | 113.96 (11) | C3—C16—H16C | 109.5 |
| N2—C8—C9 | 122.36 (12) | H16A—C16—H16C | 109.5 |
| N2—C8—H8A | 118.8 | H16B—C16—H16C | 109.5 |
| C9—C8—H8A | 118.8 | | |

| | | | |
|---|---|---|---|
| C7—N1—N2—C8 | −178.10 (14) | C5—C6—C7—N1 | −45.73 (18) |
| C6—C1—C2—C3 | −1.1 (2) | C1—C6—C7—N1 | 134.90 (14) |
| C15—C1—C2—C3 | −178.87 (15) | N1—N2—C8—C9 | −179.45 (13) |
| C1—C2—C3—C4 | 2.2 (2) | N2—C8—C9—C10 | −163.72 (16) |
| C1—C2—C3—C16 | −177.21 (15) | N2—C8—C9—C14 | 17.1 (2) |
| C2—C3—C4—C5 | −1.7 (2) | C14—C9—C10—C11 | −1.5 (2) |
| C16—C3—C4—C5 | 177.66 (15) | C8—C9—C10—C11 | 179.34 (15) |
| C3—C4—C5—C6 | 0.2 (2) | C9—C10—C11—C12 | 0.6 (3) |
| C4—C5—C6—C1 | 0.9 (2) | C10—C11—C12—O3 | −178.29 (14) |
| C4—C5—C6—C7 | −178.49 (13) | C10—C11—C12—C13 | 0.6 (2) |
| C2—C1—C6—C5 | −0.5 (2) | O3—C12—C13—O2 | −2.27 (19) |
| C15—C1—C6—C5 | 177.23 (14) | C11—C12—C13—O2 | 178.79 (14) |
| C2—C1—C6—C7 | 178.88 (13) | O3—C12—C13—C14 | 178.14 (12) |
| C15—C1—C6—C7 | −3.4 (2) | C11—C12—C13—C14 | −0.8 (2) |
| N2—N1—C7—O1 | −5.9 (2) | O2—C13—C14—C9 | −179.69 (13) |
| N2—N1—C7—C6 | 172.84 (12) | C12—C13—C14—C9 | −0.1 (2) |
| C5—C6—C7—O1 | 133.03 (16) | C10—C9—C14—C13 | 1.3 (2) |
| C1—C6—C7—O1 | −46.3 (2) | C8—C9—C14—C13 | −179.59 (14) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1*W*—H1*W*1···N2$^{i}$ | 0.87 (2) | 2.20 (2) | 3.059 (2) | 169 (2) |
| N1—H1*A*···O3$^{ii}$ | 0.91 (2) | 2.08 (2) | 2.962 (2) | 163 (2) |
| O1*W*—H2*W*1···O1 | 0.94 (2) | 2.01 (2) | 2.935 (2) | 173 (2) |
| O2—H2*A*···O1$^{i}$ | 0.88 (2) | 1.94 (2) | 2.791 (2) | 162 (2) |
| O3—H3*A*···O1*W*$^{iii}$ | 0.85 (2) | 1.79 (2) | 2.629 (2) | 172 (2) |
| C8—H8*A*···O3$^{ii}$ | 0.93 | 2.58 | 3.382 (2) | 145 |
| C15—H15*B*···O2$^{i}$ | 0.96 | 2.52 | 3.351 (2) | 144 |

Symmetry codes: (i) −*x*, −*y*+1, −*z*; (ii) *x*+1/2, −*y*+1/2, *z*−1/2; (iii) *x*+1/2, −*y*+1/2, *z*+1/2.